PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 24 August 2000 (24.08.00)	
International application No. PCT/GB99/04425	**Applicant's or agent's file reference** JDM/P401620WO
International filing date (day/month/year) 24 December 1999 (24.12.99)	**Priority date** (day/month/year) 24 December 1998 (24.12.98)
Applicant WHITWORTH, Andrew, John	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

18 July 2000 (18.07.00)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland**	Authorized officer S. Mafla
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT OR THE DECLARATION

(PCT Rule 44.1)

To:
W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
UNITED KINGDOM

Date of mailing *(day/month/year)* 27/03/2000	
Applicant's or agent's file reference JDM/P401620WO	**FOR FURTHER ACTION** See paragraphs 1 and 4 below
International application No. PCT/GB 99/04425	International filing date *(day/month/year)* 24/12/1999
Applicant WHITWORTH, ANDREW, JOHN et al.	

1. [X] The applicant is hereby notified that the International Search Report has been established and is transmitted herewith.

Filing of amendments and statement under Article 19:
The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41–22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. [ ] The applicant is hereby notified that no International Search Report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

3. [ ] **With regard to the protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

[ ] the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

[ ] no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

Shortly after **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90*bis*.1 and 90*bis*.3, respectively, before the completion of the technical preparations for international publication.

Within **19 months** from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

Within **20 months** from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the International Searching Authority European Patent Office, P.B. 5818 Patentlaan 2 NL–2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Authorized officer Lucia Van der Leeden

Form PCT/ISA/220 (July 1998)

NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international pbulication. Furthermore, it should be emphasized that provisional protection is available in some States only.

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When? Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How? Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped),whether

(i) the claim is unchanged;

(ii) the claim is cancelled;

(iii) the claim is new;

(iv) the claim replaces one or more claims as filed;

(v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."

2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."

3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."

4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international appplication is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
JDM/P401620WO		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/GB 99/ 04425	24/12/1999	24/12/1998
Applicant		
WHITWORTH, ANDREW, JOHN et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

[ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

[ ] contained in the international application in written form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readble form.

[ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

[X] the text is approved as submitted by the applicant.

[ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

[X] the text is approved as submitted by the applicant.

[ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 1

[X] as suggested by the applicant.

[ ] None of the figures.

[ ] because the applicant failed to suggest a figure.

[ ] because this figure better characterizes the invention.

INTERNATIONAL SEARCH REPORT

[redacted]

International Application No
[redacted]GB 99/04425

LASSIFICATION OF SUBJECT MATTER
[redacted]7 F16L11/11

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 F16L B29C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passagee	Relevant to claim No.
A	EP 0 474 449 A (ANDREW JOHN WHITWORTH AND DORO) 11 March 1992 (1992-03-11) cited in the application column 5, line 46 -column 6, line 20; claims 1,2 column 2, line 21 - line 30	1-20
A	GB 1 543 586 A (WHITWORTH B) 4 April 1979 (1979-04-04) cited in the application page 3, left-hand column, line 36 - line 60; claim 1	1-20

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 March 2000	27/03/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Budtz-Olsen, A

1

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

[redacted]ational Application No
[redacted]T/GB 99/04425

ontinuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 293 222 A (JONES KENNETH NEIL JOHN ;JONES IAN CAMPBELL (GB); JONES STUART GRA) 20 March 1996 (1996-03-20) cited in the application page 17, line 36 abstract	1-20
A	US 5 476 080 A (BRUNNHOFER ERWIN) 19 December 1995 (1995-12-19) column 2, line 18 - line 25 column 2, line 34 - line 47	1-20

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 99/04425

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0474449 A	11-03-1992	AT 124520 T	15-07-1995
		DE 69110801 D	03-08-1995
		DE 69110801 T	23-11-1995
		ES 2075360 T	01-10-1995
GB 1543586 A	04-04-1979	FR 2435342 A	04-04-1980
GB 2293222 A	20-03-1996	NONE	
US 5476080 A	19-12-1995	DE 4330855 C	13-10-1994
		BR 9403479 A	16-05-1995
		ES 2119597 A	01-10-1998
		FR 2709707 A	17-03-1995
		GB 2281951 A,B	22-03-1995
		IT MI941734 A,B	13-03-1995
		JP 2718896 B	25-02-1998
		JP 7205328 A	08-08-1995

PATENT COOPERATION TREATY

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:
W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
GRANDE BRETAGNE

17 OCT 2000
LIVERPOOL

PCT

WRITTEN OPINION

(PCT Rule 66)

Date of mailing *(day/month/year)*	13.10.2000
Applicant's or agent's file reference JDM/P401620WO	**REPLY DUE** **within 3 month(s)** from the above date of mailing

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB99/04425	24/12/1999	24/12/1998

International Patent Classification (IPC) or both national classification and IPC
F16L11/11

Applicant
WHITWORTH, ANDREW, JOHN et al.

RESPONSE DUE: 13.1.01

1. This written opinion is the **first** drawn up by this International Preliminary Examining Authority.

2. This opinion contains indications relating to the following items:

I	☒	Basis of the opinion
II	☐	Priority
III	☐	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
IV	☐	Lack of unity of invention
V	☒	Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
VI	☐	Certain document cited
VII	☐	Certain defects in the international application
VIII	☒	Certain observations on the international application

3. The applicant is hereby **invited to reply** to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also: For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.

4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 24/04/2001.

Name and mailing address of the international preliminary examining authority:
European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer / Examiner
Kujat, C

Formalities officer (incl. extension of time limits)
Nilles, F
Telephone No. +49 89 2399 2931

I. Basis of the opinion

1. This opinion has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*):

Description, pages:

1-20 as originally filed

Claims, No.:

1-20 as originally filed

Drawings, sheets:

1/4-4/4 as originally filed

2. The amendments have resulted in the cancellation of:

☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	19-20
Inventive step (IS)	Claims	
Industrial applicability (IA)	Claims	

2. Citations and explanations

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following document:

D1: EP 0.474.449

Independent Claim 19:

1.1 Document D1, which is considered being the closest prior art for independent claim 19, discloses a
PTFE (column 2, line 5) tube comprising external roots and peaks (column 2, lines 9-11) which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1 (column 2, lines 21-26) and W_1 is less than 25% of W_0 (column 4, lines 31-35).

1.2 The subject matter of claim 19 therefore lacks novelty over D1.

Dependent Claim 20

2. Dependent claim 20 does not contain any features which, in combination with the features of claim 19, meet the requirements of the PCT in respect of novelty, since the additional feature of dependent claim 20 is also disclosed in D1 (column 4, lines 31-35).

Re Item VIII

Certain observations on the international application

3.1. Contrary to Rule 10.2, in claim 1 two different expressions are used for the same

feature: "internal diameter ID" and "nominal bore ID".

3.2. On page 2, a value for " flexural modulus" is given, but no unit for this value is indicated.

3.3 On page 13, two values for "leakage rate" are given. The unit is wrongly indicated "cc per hour". Since leakage is also dependent on the surface, the correct unit is "cc per hour per meter" (in case of a cylindrical sample).

3.4 Minor editorial mistakes have been found in the description:
a) page 1: "of course..." instead of "Of course...".
b) page 2: "...by processes which concertina..."
c) page 5: Twice, "...tensile..." instead of "...tensile strength..."

amend page: 2
13
1
2
5
21
24

14

PATENT COOPERATION TREATY

RECD 24 JAN 2001

WIPO

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
JDM/P401620WO		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB99/04425	24/12/1999	24/12/1998
International Patent Classification (IPC) or national classification and IPC F16L11/11		
Applicant WHITWORTH, ANDREW, JOHN et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 6 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 6 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
18/07/2000	19.01.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Kujat, C Telephone No. +49 89 2399 2360

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/04425

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*:
Description, pages:

3,4,6-12,14-20	as originally filed			
1,2,5,13	as received on	22/12/2000	with letter of	15/12/2000

Claims, No.:

6 (part),7-17, 18 (part)	as originally filed			
1-5,6 (part), 18 (part)	as received on	22/12/2000	with letter of	15/12/2000

Drawings, sheets:

1/4-4/4	as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   - ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
   - ☐ the language of publication of the international application (under Rule 48.3(b)).
   - ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   - ☐ contained in the international application in written form.
   - ☐ filed together with the international application in computer readable form.
   - ☐ furnished subsequently to this Authority in written form.
   - ☐ furnished subsequently to this Authority in computer readable form.
   - ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   - ☐ The statement that the information recorded in computer readable form is identical to the written sequence

listing has been furnished.

4. The amendments have resulted in the cancellation of:

☐ the description, pages:

☒ the claims, Nos.: 19,20

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-18
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-18
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-18
	No:	Claims	

2. Citations and explanations
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

D1: EP 0.474.449
D2: US 5.476.080

Independent Claim 1:

1.1 Document D1, which is considered being the closest prior art, discloses a PTFE tube according to the preamble of claim 1. The subject matter of claim 1 differs from D1 in that the convoluted PTFE tube has an improved resistance to permeation of greater than 7.6% by comparison with the non-convoluted tube, the comparison being made between tubes of (i) equal nominal bore and (ii) equal weight of PTFE per unit length.

1.2 Since the improved resistance to permeation is given in relation to a tube obtained from a conventional process, it becomes clear that said feature is a direct and unambiguous consequence of the process of post treatment of the tube. The post treatment is implied by the comparison made. Document D2 disclosing also a PTFE tube having improved resistance to permeation does not teach post treatment, but an additional layer of plastics instead.

1.3 The subject matter of claim 1 therefore involves an inventive step.

1.4 Since the improved resistance to permeation is given in relation to a tube obtained from a conventional process, it is further not necessary to specify in the claim the method according to which permeation is measured.

Dependent claims 2-6:

2. Claims 2-6 are dependent on claim 1 and as such also meet the requirements of the

PCT with respect to novelty and inventive step.

Independent Claim 7:

3.1 Document D1, which is considered being the closest prior art, discloses a PTFE tube according to the preamble of claim 7. The subject matter of claim 7 differs from D1 in that the tube is obtained from a process comprising:
a) subjecting the PTFE tube to a deformation force at a temperature at or above the gel transition temperature of PTFE to produce constrained convolutions having a thinned wall W1 and
b) cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W1 until the convolutions having the thinned wall W1 have become stable.

3.2 These distinguishing features imply a modification of properties (morphology) by heat treatment. The problem to be solved by the present invention may therefore be regarded as rendering the tube flexible and increasing its resistance to permeation.

3.3 Departing from the teaching of D1, it is not obvious to include said process steps a) and b), as none of the documents in the International Search Report discloses or implies such features related to permeation.

Dependent claim 8:

4. Claim 8 is dependent on claims 1 and 7 and as such also meets the requirements of the PCT with respect to novelty and inventive step.

Independent Claim 9:

5. Independent claim 9 relates to a process which inevitably results in the manufacture of the product according to independent claims 1 or 7. Therefore, independent claim 9 also involves an inventive step.

Dependent Claims 10-14:

6. Claims 10-14 are dependent on claim 9 and as such also meet the requirements of the PCT with respect to novelty and inventive step.

Independent Claims 15-18:

7. Independent claims 15-18 disclose the use of the product according to independent claims 1 and 7. Therefore, independent claims 15-17 also involve an inventive step.

DESCRIPTION

A PTFE TUBE

The present invention relates to a polytetrafluoroethylene (PTFE) tube and more particularly to a PTFE tube for a flexible hose. In particular the invention relates to a PTFE tube having a smooth bore for use in the production of a lined hose assembly further comprising hose braids, external hose protection and end fittings.

It should be noted that there are two basic types of internal tube configuration;

smooth bore tubes, as their name suggests, have a substantially convolution free internal surface;

in contrast, internally convoluted tubes, as their name suggests, comprise a number of distinct peaks and roots.

Of course smooth bore tubes are often not totally devoid of bumps and indentations and may show rippling. This is however in sharp contrast to the induced peaks and roots of an internally convoluted tube.

PTFE is a unique material and is favoured for applications in the transport of foodstuffs and chemicals because of its chemical resistance and non-stick nature. However PTFE is not naturally elastic.

Producing a flexible PTFE tube for certain applications, particularly high pressure applications, where fluids, more particularly gases and vapours, are pumped through the tube has proved difficult. Indeed, it had previously been

thought that many convoluted PTFE tubes would not be suitable for such applications because the "thinning" of the walls to produce "flex" was expected to result in increased permeation to fluids.

To reduce permeation one or more of the following techniques have hitherto been employed:

1. Wall thicknesses have been increased;
2. Higher grade polymers have been used; or
3. Polymers have been processed to have increased crystalinity.

Increasing the wall thickness decreases the flexibility of the finished product as well as increasing its weight and cost.

Increasing crystalinity increases the flexural modulus of the material thus decreasing the flexibility and this also incurs a reduction in flex life.

Du Pont, for example, define crystalinity as being low (50%), moderately high (72%) or very high (82%). At low crystalinity the product has a flexural modulus of 54,000 psi and a relative permeability to $C0_2$ gas of 6; at moderately high crystalinity the product has a flexural modulus of 150,000 psi and relative permeability to CO_2 gas of 0.8 and at a very high crystalinity the product has a flexural modulus of 170,000 psi and a relative permeability to $C0_2$ gas of 0.2.

Most corrugated products are made by a process which convolutes or concertinas the product and have walls which are substantially uniform in thickness throughout. Typical processes include those described in GB 1543586 and GB 2293222.

having a thinned wall W_1; and

2. cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W_1 until the convolutions having the thinned wall W_1 have become stable.

Preferably W_1 is less than 25% of W_0.

More preferably W_1 is about 20% of W_0. In a preferred embodiment the PTFE tube is produced on a mandrel of substantially the same size as the internal diameter of a plane cylindrical PTFE paste extruded tube such that the resulting tube is a smoothbore, externally convoluted, tube. The resulting smoothbore tube has a rippled appearance.

That the deformation has become stable can be characterised by an increase in tensile strength indicating that the deformation is a "yield" deformation. The deformation can be further characterised in that it is reversible. i.e. when the deformed material is reheated to at or above the gel transition temperature without a restraining force in place, it returns substantially to its original form.

It is also possible to determine whether or not the PTFE was deformed at a temperature above or below the gel transition temperature. A tube deformed below the gel transition temperature will revert partially or substantially to its original form at temperatures below the gel transition temperature whereas one deformed at or above the gel transition temperature will only revert substantially to its original form at or above the gel transition temperature.

The increase in tensile strength can be seen by conducting a simple test. A

resulting from the processing of sample 1 in accordance with the methodology described with reference to Fig. 1 over braided with steel, end fittings swaged at each end.

Test results

Sample 1 leakage rate 220 cc per hour per metre.

Sample 2 leakage rate 150 cc per hour per metre.

Since the weight of the tube per unit length in sample 2 was approximately 20% less than the weight of the material per unit length of sample 1, the figures were adjusted to give a figure for a tube of a given weight.

Thus the specific improvement in permeation resistance is

$$\frac{220}{150} \times \frac{5}{4} = 1.83$$

in other words, the specific permeation has been reduced in the ratio of 1:0.55. Furthermore the flexibility is improved. In this regard sample 1 kinks at a bend radius of 381mm whereas sample 2 kinks at a bend radius of 63.5mm.

In a further test a comparison was made between a smoothbore externally convoluted tube made by the method of the invention and one made entirely at a temperature below 327°C.

The results of the comparison are given below:

Sample 3 (Plain tube). Leakage Rate 241cc/h/m

Sample 4 (Externally convoluted tube processed below 327°C) Leakage rate 224cc/h/m.

CLAIMS

1. A PTFE tube comprising external roots and peaks, which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 and an internal diameter ID by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1, characterised in that the convoluted PTFE tube has an improved resistance to permeation of greater than 7.6% by comparison with the non-convoluted tube, the comparison being made between tubes of (i) equal internal diameter ID; and (ii) equal weight of PTFE per unit length.

2. A PTFE tube as claimed in claim 1 wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 10%.

3. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 20%.

4. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 30%.

5. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 60%.

6. A PTFE tube as claimed in any preceeding claim having a smooth internal

assembly as claimed in claim 15.

PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

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NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing *(day/month/year)*	19.01.2001
Applicant's or agent's file reference: JDM/P401620WO	**IMPORTANT NOTIFICATION**

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB99/04425	24/12/1999	24/12/1998

Applicant
WHITWORTH, ANDREW, JOHN et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

   The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

   Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

   For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

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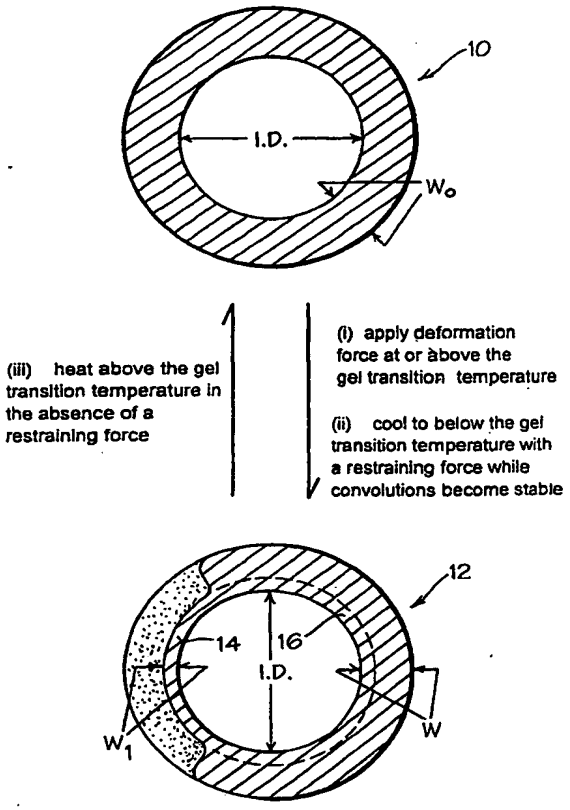
(54) Title: A PTFE TUBE

(57) Abstract

The present invention relates to a polytetrafluoroethylene (PTFE) tube, and more particularly to a PTFE tube for a flexible hose. In particular the invention relates to a PTFE tube having a smooth bore for use in the production of a lined hose assembly further comprising hose braids, external hose protection and end fittings. The PTFE tube comprises external roots and peaks, which tube is obtainable from a non–convoluted tube having an original wall thickness W_0 and an internal diameter ID by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1 characterised in that the convoluted PFTE tube has an improved resistance, of greater than 7.6 %, to permeation by comparison with the non–convoluted tube, the comparison being made between tubes of (i) equal nominal bore ID; and (ii) equal weight of PTFE per unit length.



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DESCRIPTION

A PTFE TUBE

The present invention relates to a polytetrafluoroethylene (PTFE) tube and more particularly to a PTFE tube for a flexible hose. In particular the invention relates to a PTFE tube having a smooth bore for use in the production of a lined hose assembly further comprising hose braids, external hose protection and end fittings.

It should be noted that there are two basic types of internal tube configuration;

smooth bore tubes, as their name suggests, have a substantially convolution free internal surface;

in contrast, internally convoluted tubes, as their name suggests, comprise a number of distinct peaks and roots.

of course smooth bore tubes are often not totally devoid of bumps and indentations and may show rippling. This is however in sharp contrast to the induced peaks and roots of an internally convoluted tube.

PTFE is a unique material and is favoured for applications in the transport of foodstuffs and chemicals because of its chemical resistance and non-stick nature. However PTFE is not naturally elastic.

Producing a flexible PTFE tube for certain applications, particularly high pressure applications, where fluids, more particularly gases and vapours, are pumped through the tube has proved difficult. Indeed, it had previously been

thought that many convoluted PTFE tubes would not be suitable for such applications because the "thinning" of the walls to produce "flex" was expected to result in increased permeation to fluids.

To reduce permeation one or more of the following techniques have hitherto been employed:

1. Wall thicknesses have been increased;
2. Higher grade polymers have been used; or
3. Polymers have been processed to have increased crystalinity.

Increasing the wall thickness decreases the flexibility of the finished product as well as increasing its weight and cost.

Increasing crystalinity increases the flexural modulus of the material thus decreasing the flexibility and this also incurs a reduction in flex life.

Du Pont, for example, define crystalinity as being low (50%), moderately high (72%) or very high (82%). At low crystalinity the product has a flexural modulus of 54,000 and a relative permeability to $C0_2$ gas of 6; at moderately high crystalinity the product has a flexural modulus of 150,000 and relative permeability to CO_2 gas of 0.8 and at a very high crystalinity the product has a flexural modulus of 170,000 and a relative permeability to $C0_2$ gas of 0.2.

Most corrugated products are made by processes which concertina the product and have walls which are substantially uniform in thickness throughout. Typical processes include those described in GB 1543586 and GB 2293222.

EP 474449 B1 on the other hand discloses a corrugated plastics tube which has been subject to a compression force to displace material in the root region. It is characterised in that the compression force applied was sufficient to take the plastics of the tube, which was at a temperature below its melt temperature, beyond its elastic point. This can be achieved at any temperature below the melt temperature and the patent makes no specific teaching in this regard. Furthermore, the patent relates to plastics in general and is directed to producing flexibility. It is not particular to PTFE (although PTFE is specified) and it does not address the problem of producing tubes with improved permeability resistance to gases.

In contrast the present invention, which is particular to PTFE, teaches that a novel product is obtained by a process comprising

1. subjecting the PTFE tube to a deformation force at a temperature at or above the gel transition temperature of PTFE to produce constrained convolutions having a thinned wall W_1; and

2. cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W_1 until the convolutions having the thinned wall W_1 have become stable.

This product is characterised in that the convoluted PFTE tube has an improved resistance, of greater than 7.6%, to permeation by comparison with the non-convoluted tube, the comparison being made between tubes of (i) equal nominal bore ID; and (ii) equal weight of PTFE per unit length.

This improved resistance to permeation is indicative of the fact the product

processed in this manner has a different form to one not so processed. This can be confirmed by way of the test procedure set out in the specific description.

Surprisingly, the applicants have discovered that by processing the PTFE, which term includes modified PTFE, in a particular manner they are able to reduce permeation rates for a given thickness of PTFE. That the PTFE processed in this manner has a changed form can be characterised by amongst other things, its improved resistance to permeation and increased tensile strength.

According to a first aspect of the present invention there is provided a PTFE tube comprising external roots and peaks which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 and an internal diameter ID by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1, characterised in that the convoluted PTFE tube has an improved resistance to permeation of greater than 7.6% by comparison with the non-convoluted tube, the comparison being made between tubes of (i) equal nominal bore ID; and (ii) equal weight of PTFE per unit length.

Preferably the PTFE tube has a smooth internal bore.

In one embodiment the smoothbore has a rippled appearance.

According to a further aspect of the present invention there is provided a method of producing a PTFE tube comprising external roots and peaks from a non-convoluted tube having an original wall thickness W_0 comprising:

1. subjecting the PTFE tube to a deformation force at a temperature at or above the gel transition temperature of PTFE to produce constrained convolutions

having a thinned wall W_1; and

2. cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W_1 until the convolutions having the thinned wall W_1 have become stable.

Preferably W_1 is less than 25% of W_0.

More preferably W_1 is about 20% of W_0.

In a preferred embodiment the PTFE tube is produced on a mandrel of substantially the same size as the internal diameter of a plane cylindrical PTFE paste extruded tube such that the resulting tube is a smoothbore, externally convoluted, tube. The resulting smoothbore tube has a rippled appearance.

That the deformation has become stable can be characterised by an increase in tensile indicating that the deformation is a "yield" deformation. The deformation can be further characterised in that it is reversible. i.e. when the deformed material is reheated to at or above the gel transition temperature without a restraining force in place, it returns substantially to its original form.

It is also possible to determine whether or not the PTFE was deformed at a temperature above or below the gel transition temperature. A tube deformed below the gel transition temperature will revert partially or substantially to its original form at temperatures below the gel transition temperature whereas one deformed at or above the gel transition temperature will only revert substantially to its original form at or above the gel transition temperature.

The increase in tensile can be seen by conducting a simple test. A

longitudinal section is taken from a convoluted tube prepared in accordance with the invention and is gripped on either side of a root. It is then pulled apart until the section breaks at the root. By first determining the thickness and width of the root and noting the force applied to break the tube at its root the breaking force per cross sectional area can be calculated. Another section of the tube is then heated to above the gel transition temperature so it reverts to its starting conformation and the section is then subjected to the same test i.e. it is pulled along the longitudinal axis of the tube. Typical results obtained will be 41368 kPa (6000psi) for a plain tube and 75842 kPa (11000 Psi) for a convoluted tube manufactured in accordance with the invention.

The permeability properties of PTFE tubes deformed in this manner were totally unexpected as a product which was more permeable was expected as a consequence of a "thinning" of the walls.

For the avoidance of doubt the term gel transition temperature as used herein refers to the temperature at which PTFE becomes more transparent and amorphous. This is at a temperature of between 325°C and 340°C and is generally considered to be at a temperature of 327°C. This temperature is sometimes inappropriately, in a processing context, referred to as the melt temperature, see for example D.I. McCaine "Co-polymers with hexafluoropropylene" see page 630. The true "melt" temperature is the temperature at which the polymer melts from its gel state to form a liquid at which point it also begins to degrade and evaporate rapidly. This is at a temperature of above 550°, approaching "red heat", see for

example R.J. Plunkett the inventor of PTFE.

Without wishing to be bound by theory it is believed that at temperatures above 327°C a given applied deformation force is less likely to cause "cut" than the same deformation applied at temperatures below 327°C. Furthermore because the material is elastically deformed as opposed to being "cut" it benefits from improved characteristics, for example, improved resistance to permeability and increased tensile strength. These characteristics show themselves in the convoluted tubes ability to revert substantially to its original form on re-heating to above 327°C without a restraining force in place. The greater the "cutting" during processing the greater the depth of any "nicks" which appear in the so reverted product and the less it will resemble its original form.

At processing temperatures below 327°C the deformations will include, for a critical force, deformations beyond the products elongation break point which will not repair. Only deformations beyond yield, and not those beyond the products elongational break point will revert to their original shape on re-heating to above 327°C. "Cutting" can, of course, also occur at temperatures above the gel transition temperature if the deformation caused by the force is sufficient. The critical deformation will, however, be less at a temperature of below 327°C. For example, a smooth bore convoluted tube processed at below the gel transition temperature will, above a critical deformation, exhibit significant cut. Below this critical deformation a tube can only be thinned in the root region to between one third to one quarter of its original thickness. When processing at temperatures

above the gel transition temperature, the tube can be thinned to about one fifth of its original thickness without exhibiting cutting.

Thus, according to another aspect of the present invention there is provided a PTFE tube comprising external roots and peaks, which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1 characterised in that W_1 is less than 25% of W_0.

Preferably W_1 is about 20% of W_0.

The term "returns substantially to its original form" is intended to mean that the reverted tube does not have significant convolutions, although it may show signs of limited damage caused by deformations beyond elongation at break point in the form of cuts or nicks. The product will, however, return to within 20%, more preferably 10% and more preferably still 5% of its original wall thickness W_0.

Because the force applied to the tube to form roots is 3-dimensional it cannot readily be determined. However, the deformation can be measured as indicated above. As a general rule greater deformations can be achieved without cutting at higher temperatures. Above 327°C deformation without cutting is about 20% better than below 327°C as indicated by the greater thinning which can be achieved when processing at temperatures above the gel transition temperature. Of course the deformations can not be fixed above 327°C therefore to fix the deformations a restraining force needs to be maintained whilst the temperature is

dropped to below 327°C such that the deformations become stable.

The invention will now be described, by way of example only, with reference to Figs 1 to 6 in which:

Fig. 1 is a schematic diagram showing the reversable nature of the production of a PTFE tube according to the invention;

Fig. 2 which is an enlarged sectional view of a segment of a PTFE tube comprising external roots and peaks and a smooth internal bore;

Fig. 3 is a cut-away view of a hose assembly comprising a (liner) tube according to the invention;

Fig. 4 is a graph showing maximum working pressure vs temperature for different sized PTFE tubes according to the invention;

Fig. 5 is a graph of flow rate vs pressure drop for different sized tubes according to the invention; and

Fig. 6 is a diagram of an apparatus used for conducting the permeability test.

The invention is further illustrated with reference to a table, which shows the specification of a number of different sized tube and hose assemblies.

Finally, examples with comparative data, showing the improved permeability resistance of a tube processed in accordance with the invention are given.

Referring to Fig. 1 a plane cylindrical PTFE paste extruded tube 10 with a internal diameter I.D. of 25.4 mm and a thickness W_0 of 2.29 mm was heated

to 380°C on a suitably sized mandrel. A tool with an internal diameter which was greater than the outside diameter of the mandrel but less than the combined diameter of the outside diameter of the mandrel and twice the wall thickness W_0 of the plastic tube was bought into contact with the tube so that its leading end applied a pressure sufficient to displace material to form a smooth bored convoluted tube 12 comprising roots 14, (with a root wall thickness W_1) and peaks 16 (with a peak wall thickness W) whilst maintaining the tube at a temperature above the gel transition temperature. The following end of the tool was maintained at a temperature below the gel transition temperature such that the following end of the tool cooled the convoluted tube to below the gel transition temperature whilst applying a restraining force such that the convolutions became stable. The helical tool was rotated relative to the mandrel at a speed of 18 revs per minute such that the leading end applied a 3-dimensional deformation force at above the gel transition temperature and the following end applied a restraining force until the temperature had dropped below the gel transition temperature and the convolutions had become stable. In this regard, each section of the convoluted tube was subjected to the restraining forces within the tool for approximately 1 minute.

Different shapes can of course be produced using the method. In one embodiment a single start spiral corrugation may be formed. Alternatively multiple start spiral corrugations, annular corrugations, axial corrugations or a combination thereof can be produced.

The root wall should be thinned from 60% to 5%, preferably 50% to 5%,

of its original value Wo. For a fully corrugated shape the root wall should preferably be thinned to 40% to 20% or its original value and for a smooth bore shape to 30% to 20%.

Preferably a radiussed rather than square edge to the thinned region is formed.

As far as the width of the thinned area is concerned, working from original wall thickness Wo, the width should be 10% - 200% of the peak wall thickness W for smooth bore type constructions and typically 30 - 600% of peak wall thickness for fully corrugated constructions. As the width tends to a greater percentage so the flexibility of the product increases.

The PTFE tube 12 has a root wall thickness W_1 which is less than the peak wall thickness W, when the root wall thickness W_1 has been reduced during construction from a standard wall thickness W_0 by compressing the tube to displace material.

Furthermore compression and displacement can cause the peak wall thickness W to be greater than the original wall thickness W_O.

As illustrated in Fig. 1 the resulting tube can be returned to its starting form by reheating the tube 12 to above its gel transition temperature without a restraining force in place.

RESULTS OF PERMEABILITY TESTS ON THE TUBES OF SAMPLE 1 AND SAMPLE 2

Two sample tubes were subjected to a permeability test using an apparatus

as illustrated in Fig. 6. The apparatus comprises a helium supply 50, a pressure regulator 52, a connecting tube 54 and a bleed valve 58. The test sample 56 is connected between the connecting tube 54 and the bleed valve 58. The test sample is immersed horizontally in a water bath 60 and a collecting cowl 62 and calibrating column 64 positioned thereover. The samples 56, which are known lengths of PTFE tubing, are overbraided with end fittings swaged at both ends.

Prior to testing, the apparatus is first purged ensuring only helium remains in the sample (it is held vertically with the bleed valve the lower end). The sample is then immersed in the water bath, supported horizontally, and the helium pressure is increased to the test pressure. The apparatus is left for a minium of 30 minutes to allow steady state permeation to be achieved. The collecting cowl is placed over the sample, with water filling both it and the calibrated collecting column. Collection of all permeating gas is timed and the amount recorded. The procedure is repeated several times to ensure steady state permeation has been achieved and the results are reproducible.

Test Conditions

Commercial grade Helium at 29.6 At (30 Bar) at room temperature.

Leakage is determined after steady state permeation has been reached on the samples as follows:

Sample 1. Plain cylindrical tube with an internal diameter of 25.4mm and a wall thickness of 2.29mm over braided with steel, end fittings swaged at each end.

Sample 2. Smooth bore convoluted tube with an internal diameter of 25.4 mm

resulting from the processing of sample 1 in accordance with the methodology described with reference to Fig. 1 over braided with steel, end fittings swaged at each end.

Test results

Sample 1 leakage rate 220 cc per hour.

Sample 2 leakage rate 150 cc per hour.

Since the weight of the tube per unit length in sample 2 was approximately 20% less than the weight of the material per unit length of sample 1, the figures were adjusted to give a figure for a tube of a given weight.

Thus the specific improvement in permeation resistance is

$$\frac{220}{150} \times \frac{5}{4} = 1.83$$

in other words, the specific permeation has been reduced in the ratio of 1:0.55. Furthermore the flexibility is improved. In this regard sample 1 kinks at a bend radius of 381mm whereas sample 2 kinks at a bend radius of 63.5mm.

In a further test a comparison was made between a smoothbore externally convoluted tube made by the method of the invention and one made entirely at a temperature below 327°C.

The results of the comparison are given below:

Sample 3 (Plain tube). Leakage Rate 241cc/h/m

Sample 4 (Externally convoluted tube processed below 327°C) Leakage rate 224cc/h/m.

Sample 5 (Externally convoluted tube processed above 327°C and below 550°C and cooled below 327°C with a restraining force). Leakage Rate 148cc/hr/m.

Figures are permeation rates of helium at 29.6 At (30 Bar) at room temperature.

Below 327°C there is an apparent improvement of 7.6% whereas at above 327°C there is a very significant improvement of 62.8%.

In practice it has been found that best results are achieved when the temperature is between 327°C - 450°C, more preferably 327°C - 420°C , since deformation is achieved without straining the material beyond the elongation break limit in any position in the convoluted configuration. The elongation limit increases with the processing temperature.

Whilst the invention has been specifically described with reference to a smooth bored convoluted tube, it will be apparent to the skilled man that convoluted tubes of various configurations can benefit from the method of the invention.

A tube as outlined above, particularly a smoothbore with external convolution, has many applications since it overcomes the disadvantages of either conventional smooth bore or internally and externally convoluted flexible hose designs, dramatically improving on many of their individual technical performance parameters.

The tube can be used as a hose liner in a hose assembly. It comprises (see Fig. 2) integral rib sections (peaks) 16 which support the tube against kinking, vacuum and pressure and highly compressed web sections (roots) 14 leaving a

smoothbore inner surface 18 which is rippled and provides excellent flexibility.

A hose comprising a smoothbore externally convoluted PTFE tube according to the invention shows significant improvement in properties when compared to a conventional convoluted hose (both internal and external convoluted). These include:

Vastly improved hygienic cleanability due to its smoothbore and polished surface finish;

Flow Rates which are more than 100% higher.

Pressure Ratings which are more than 50% higher.

Gas Permeation Resistance which is more than 150% higher; and

Flex Life at Temperature and Pressure which is more than 50 times the life (dependent upon test conditions).

One type of hose comprising a PTFE tube according to the invention, is illustrated in Fig. 3. It comprises a PTFE liner tube 20, with external convolutions 22 of peaks 24 and roots 26 and a smooth internal bore 28 with slight ripples 30. The inner surface has been hot polished. The liner tube has a flared end 32. The external surface of the liner is covered with a braid 34 over most of its length. Attached to the ends of the tube are end fittings 36. A spigot 38 lies between the tube and braid and is secured by a ferrule 40.

The PTFE (line tube is either made from FDA approved PTFE (hose grade) or is made antistatic by the inclusion of, for example, carbon black.

The former is, for use in all applications where fluids conveyed are not highly

electrically resistant. The latter is suitable for use in applications where electrically resistive fluids, such as fuels, solvents or freons are being conveyed. The antistatic nature of the tube prevents a damaging electrostatic charge build-up inside the hose.

The unique properties of the smoothbore, externally convoluted tubes make them suitable for use in circumstances where conventional PTFE lined hoses would not be suitable.

In particular, hoses with a 15 mm - 50 mm hose bore size can be used at full vacuum up to 130°C. Above this the vacuum resistance should be reduced 1% for every degree over 130°C. Fig. 4 shows the relationship between maximum working pressure (in Bars) vs temperature in °C for a range of hose sizes.

Flow rates are also greater than for conventional convoluted PTFE hose. In a straight configuration, using water as a test medium, flow rates as illustrated in Fig. 5 can be achieved.

In practice, flow rates will vary with hose flexing, fluid viscosity, end fitting design and other parameters, but in general hose flow rates 2-3 times better than conventional convoluted PTFE hose were achieved.

The specification of typical products are illustrated in Table 1 below.

Nominal Hose Bore Size mm	Actual Bore Size mm	O/D of Tube mm	Braid Type	O/D of Braid of Rubber mm	Min. Bend Radius mm	Maximum Working Pressure Bar	Minimum Burst Pressure Bar	Maximum Continuous Hose Length Mtrs	Weight per Unit Length Kg/Mtr
15	12.7	15.4	TO	-	60	10	40	20	.15
			SS	16.5	38	70	280	20	.29
			PB	18.0	38	35	140	20	.22
			SS,RC/FP/SI	22.0	60	70	280	20	.39
20	19.0	23.0	TO	-	75	10	40	20	.20
			SS	24.4	50	60	240	20	.40
			PB	25.8	50	30	120	20	.28
			SS,RC/FP/SI	30.0	75	60	240	20	.55
25	25.4	30.5	TO	-	110	8	30	20	.36
			SS	31.9	70	50	200	20	.63
			PB	34.3	70	25	100	20	.47
			SS,RC/FP/SI	38.0	110	50	200	20	.92
32	32	38.3	TO	-	130	6	24	20	.45
			SS	39.7	85	45	180	20	.85
			PB	42.1	85	23	90	20	.72
			SS,RC/FP/SI	45.7	130	45	180	20	1.15
40	38	45.0	TO	-	160	5	20	17	.66
			SS	46.8	100	40	160	17	1.10
			PB	48.8	100	20	80	17	.90
			SS,RC/FP/SI	52.8	160	40	160	17	1.55
50	50.8	58.4	TO	-	230	3	12	10	1.25
			SS	60.2	140	30	120	10	1.90
			PB	62.2	140	15	60	10	1.60
			SS,RC/FP/SI	66.2	230	30	120	10	2.56

Maximum Operating Temperatures: SS Braid -70°C to +260°C, PB Braid -30°C to +90°C. SS, RC and SS, FP -40°C to +120°C, SS,SI -40° to +180°C.

Pressure Variation with Temperature: SS Braid as per Fig. 4; PB pressure as above over whole temperature range; RC, FP and SI grades are per Fig. 4, BUT only within the temperature range for the particular grade.

Key.

TO tube only

SS Stainless Steel

PB Polypropylene

RC Rubber covered

FP Fire proof

SI Silicon rubber covered.

A PTFE tube may be used as a lightweight hose per se, in applications where working pressures are low and where there is no need for the physical protection offered by an external braid.

Stainless steel (SS) braided hose is used in applications involving high temperatures and working pressures. High tensile grade 304 stainless steel wire is used, to give maximum pressure resistance and external protection to the hose.

Polypropylene braided (PB) hose is often preferred in application involving frequent handling and movement of the hose, and where temperatures are within the range -30°C and +90°C. PB braid is lighter in weight, and any broken strands will not cut the operator's hands. In addition, PB braid is not prone to chloride stress corrosion.

Additional external protection may be provided over the braid.

For the most rugged applications where the hose may be subjected to rough treatment and severe external abrasion a rubber covering is provided it may also be added for hygienic applications, where external smoothness and cleanability of the hose is of prime importance.

The preferred rubber is EPDM which has excellent chemical resistance, and is temperature resistant up to 120°C.

Other external protection which may be used includes fireproof rubber; silicon rubber (resistant up to 180°C and clear); scuffrings and protection coils.

A wide range of fittings may be incorporated to form a hose. They include swivel flux fittings (as illustrated in Fig. 3) or DIN 11851 fittings (male and

female); cam action fittings, frictioned fittings and SMS and RTT fittings.

CLAIMS

1. A PTFE tube comprising external roots and peaks, which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 and an internal diameter ID by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1, characterised in that the convoluted PTFE tube has an improved resistance to permeation of greater than 7.6% by comparison with the non-convoluted tube, the comparison being made between tubes of (i) equal nominal bore ID; and (ii) equal weight of PTFE per unit length.

2. A PTFE tube as claimed in claim 1 wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 10%.

3. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 20%.

4. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 30%.

5. A PTFE tube as claimed in any preceeding claim wherein the improved resistance to permeation by comparison with the non-convoluted tube is greater than 60%.

6. A PTFE tube as claimed in any preceeding claim having a smooth internal

bore.

7. A PTFE tube as claimed in any preceeding claim, which tube is obtained from a non-convoluted tube having an original wall thickness W_0 and an internal diameter ID by a process comprising:

1. subjecting the PTFE tube to a deformation force at a temperature at or above the gel transition temperature of PTFE to produce constrained convolutions having a thinned wall W_1; and

2. cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W_1 until the convolutions having the thinned wall W_1 have become stable.

8. A PTFE tube as claimed in any preceding claim, which on heating to above its gel transition temperature without a restraining force in place returns to within 20% of the tubes original wall thickness W_0 but will not do so below the gel transition temperature.

9. A method of producing a PTFE tube comprising external roots and peaks from a non-convoluted tube having an original wall thickness W_0 comprising:

1. subjecting the PTFE tube to a deformation force at a temperature at or above the gel transition temperature of PTFE to produce constrained convolutions having a thinned wall W_1; and

2. cooling the PTFE tube to below the gel transition temperature whilst continuing to constrain the deformations having the thinned wall W_1 until the convolutions having the thinned wall W_1 have become stable.

10. A method of producing a PTFE tube as claimed in claim 9, wherein the tube is placed on a mandrel and a helical tool comprising a leading end and a following end is rotated relative to the mandrel at a speed such that the leading end applies a deformation force at above the gel transition temperature and the following end applies a restraining force until the temperature has dropped below the gel transition temperature and the convolutions have become stable.

11. A method as claimed in claim 10 wherein the mandrel is a plane cylindrical mandrel.

12. A method as claimed in claims 10 or 11 wherein the following end of the helical tool is maintained at a temperature below the gel transition temperature.

13. A method as claimed in any of claims 9 to 12 wherein W_1 is less than 25% of W_0.

14. A method as claimed in claim 13 wherein W_1 is about 20% of W_0.

15. A hose assembly comprising a PTFE tube as claimed in any of claims 1 to 8, a braid and one or more end fittings.

16. Use of a PTFE tube as claimed in any of claims 1 to 8 in a hose assembly for the purpose of improving the resistance to permeation of said hose assembly.

17. Use of a PTFE tube as claimed in any of claims 1 to 8 for the manufacture of a hose assembly intended to have improved resistance to permeation.

18. A method comprising passing a fluid through a PTFE tube or hose assembly under a pressure greater than atmospheric pressure characterised in that the fluid is passed through a PTFE tube as claimed in any of claims 1 to 8 or the hose

assembly as claimed in claim 15.

19. A PTFE tube comprising external roots and peaks which tube is obtainable from a non-convoluted tube having an original wall thickness W_0 by a process in which a region of the tube is thinned to provide external convolutions with a root wall thickness W_1 characterised in that W_1 is less than 25% of W_0.

20. A PTFE tube as claimed in claim 19 wherein W_1 is about 20% of W_0.

INTERNATIONAL SEARCH REPORT

Intern nal Application No
PCT/GB 99/04425

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 F16L11/11

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 F16L B29C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 474 449 A (ANDREW JOHN WHITWORTH AND DORO) 11 March 1992 (1992-03-11) cited in the application column 5, line 46 -column 6, line 20; claims 1,2 column 2, line 21 - line 30	1-20
A	GB 1 543 586 A (WHITWORTH B) 4 April 1979 (1979-04-04) cited in the application page 3, left-hand column, line 36 - line 60; claim 1	1-20

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search	Date of mailing of the international search report
16 March 2000	27/03/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Budtz-Olsen, A

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/04425

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

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